wherein the substituents R and R' are as aforesaid, comprises providing a solution of a silane derivative of the general formula

in an inert solvent, providing a suspension of sodium-phenyl in an inert dispersing medium in a concentration of at most 1 mol C₆H₅Na per litre, cooling the silane derivative solution to a temperature of between -20 and -80°C whilst stirring vigorously, and simultaneously adding to the silane derivative solution an amount of the suspension stoichiometrically corresponding to the desired degree of phenylation, the addition of the suspension taking place evenly over a period of at least 30 minutes, and being effected in the absence of air and water.

The silane derivative solution is suitably provided by dissolving the silane derivative in at least an equal volume of the inert solvent.

Preferably the suspension is added over

a period of 1 to 3 hours, and preferably the suspension is also cooled. The preferred temperature of the solution is between -20 and -40°C.

The sodium-phenyl suspension may be obtained in known manner from sodium and chlorobenzene, for example by introducing 92 g. of the metal in small pieces into 2 litres of toluene or another suitable dispersing agent with the exclusion of air, heating the mixture to the melting point of sodium and dispersing it, for example by means of a vibrating mixing device, allowing the mixture to cool to 10°C, adding 224 g. chlorobenzene dropwise while cooling to 10 to 15°C, and continuing the mixing for a further 1 to 2 hours. The suspension used in the Examples described below was obtained thus, and had a concentration of 0.82 mol C₆H₅Na per litre. The concentration of sodium-phenyl in the suspension can be reduced to any extent, economic consideration being the only limit.

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Depending upon the silane derivative to be phenylated and the desired silane derivative, there is used a quantity of the sodiumphenyl suspension such that 1, 2 or 3 molecules of C₆H₆Na are used for every silicon atom, as shown by the following reaction equations:—

Cl₂SiRR' + C₆H₅Na
$$\rightarrow$$
ClSi(C₆H₅)RR' + NaCl
Cl₃SiR +2 C₅H₅Na \rightarrow ClSi(C₆H₅)₂R +2 NaCl
Cl₄Si +3 C₅H₅Na \rightarrow ClSi(C₆H₅)₃ +3 NaCl

The period of time of at least 30 minutes, during which the phenylating agent is added to the chlorosilane solution, may also exceed 3 hours, but this presents no particular economic advantage.

Aromatic, aliphatic and cycloaliphatic hydrocarbons are suitable for diluting the silane derivatives, and examples of suitable diluents are benzene, toluene, xylene, hexane and cyclohexane. Solvents which are electron donors, such as dialkyl ethers, cyclic ethers and trialkylamines, can also be used for this purpose, especially in admixture with hydrocarbons.

The same solvents can be used for dispersing the sodium, provided that their boiling point is higher than the melting point of the metal (about 97°C); in the case of ethers, however, it should be noted that they may react, when in prolonged contact with sodium-phenyl, to form alcoholates.

During the process as well as during the preparation of the sodium-phenyl, the access of air or water must be excluded. It is therefore advantageous to use a protective atmosphere of inert gas, such as very pure nitrogen or argon, which may, if desired, be at superatmospheric presure. Isolation of the phenylation products is performed by known methods.

The following Examples are given for the 85 purpose of illustrating the invention.

Example 1.

292 grams diphenyl-dichlorosilane and 292 g. toluene are introduced, with the exclusion of air and moisture, into a flask fitted with stirrer, thermometer and dropping funnel. The contents of the flask are cooled externally to -20°C and 1 litre of the sodium-phenyl suspension described above is added dropwise with intense stirring over a period of 2 hours. Cooling is then interrupted and the reaction mixture is further stirred for 1 to 2 hours. The mixture is then heated to 100°C, filtered hot with the exclusion of air, the sodium. chloride remaining on the filter is washed with a little hot toluene, and the solvent is evaporated off from the combined filtrates. The desired triphenyl-chlorosilane is obtained from the residue by fractional distillation under a pressure of 0.2 mm Hg in a yield of 88% referred to the sodium-phenyl used.

EXAMPLE 2.

Using the process steps described in Example 1 a mixture of 264 g. phenyl-trichlorosilane and 264 g. toluene is reacted at -30°C with 1 litre of the sodium-phenyl suspension described above. The yield of diphenyl-di-

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chlorosilane amounts to 82% referred to the sodium-phenyl used.

EXAMPLE 3.

Again using the process described in Example 1 a mixture of 100 g. silicon tetrachloride, 675 cc dioxan and 675 cc toluene is reacted at -30°C with 1 litre of the sodium-phenyl suspension. The desired diphenyl-dichlorosilane is obtained in a yield 10 of 61.5% referred to the sodium-phenyl used. The other fractions consist of approximately equal parts by weight of phenyl-trichlorosilane and triphenyl-chlorosilane.

Example 4.

15 Again in analogy with Example 1 a mixture of 700 g. methyltrichlorosilane and 300 g. diethyl ether is reacted at -30°C with 1 litre of the sodium-phenyl suspension. The yield of phenyl-methyl-dichlorosilane amounts 20 to 78% referred to the sodium-phenyl used.

WHAT WE CLAIM IS:-

1. Proces for the controlled production of a silane derivative of the general formula

$$\sum_{\substack{Si-Ci\\R'}}^{R}$$

25 in which R and R' (same or different) each represent chlorine, a phenyl radical or an alkyl or cycloalkyl radical having 1 to 24 carbon atoms, which comprises providing a solution of a silane derivative of the general 30 formula

in an inert solvent, providing a suspension of sodium-phenyl in an inert dispersing medium in a concentration of at most 1

mol $\rm C_0 H_5 Na$ per litre, cooling the silane derivative solution to a temperature of between -20 and $-80^{\circ}\rm C$ whilst stirring vigorously, and simultaneously adding to the silane derivative solution an amount of the suspension stoichiometrically corresponding to the desired degree of phenylation, the addition of the suspension taking place evenly over a period of at least 30 minutes, and being effected in the absence of air and water.

2. Process as claimed in claim 1 wherein the silane derivative solution is provided by dissolving the silane derivative in at least an equal volume of the inert solvent.

3. Process as claimed in claim 1 or 2 wherein the silane derivative solution is cooled to a temperature of between -20 and -40°C.

4. Process as claimed in any of claims 1 to 3 wherein the addition of the suspension takes place over a period of 1 to 3 hours.

5. Process as claimed in any of claims 1 to 4 wherein the suspension is also cooled.

6. Process as claimed in any of claims 1 to 5 wherein the inert solvent is an aromatic, aliphatic or cycloaliphatic hydrocarbon, an electron donor, or a mixture thereof.

7. Process as claimed in any of claims 1 to 6 wherein the inert dispersing medium is an aromatic, aliphatic or cycloaliphatic hydrocarbon, an electron donor, or a mixture thereof, having a boiling point higher than the melting point of sodium.

8. Process as claimed in any of claims 1 to 7 wherein the reaction is carried out under an atmosphere of very pure nitrogen or argon.

9. Process for the controlled production of a silane derivative substantially as hereinbefore described in any of the Examples.

10. A silane derivative, whenever produced by a process as claimed in any of the preceding claims.

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Printed for Her Majesty's Stationery Office by the Courier Press, Learnington Spa, 1968. Published by the Patent Office, 25, Southampton Buildings, London, W.C.2, from which copies may be obtained.